SUMMARY OF THE QUALITY SYSTEMS COMMITTEE MEETING JULY 10, 2002

The Quality Systems Committee of the National Environmental Laboratory Accreditation Conference (NELAC) met on Wednesday, July 10, 2002, at 9:00 a.m., Eastern Daylight Time (EDT) as part of the Eighth Annual NELAC Meeting in Tampa, Florida. Chairperson Frederic Siegelman of the Environmental Protection Agency led the meeting. A list of Action Items is shown in Attachment A. The List of Participants is shown in Attachment B. *The purpose of the meeting was to address items of importance identified in the Agenda.*

WELCOME AND INTRODUCTIONS

Dr. Siegelman welcomed everyone to the session and called the meeting to order. He then asked all Committee members to introduce themselves. Dr. Siegelman thanked all the members of the Quality Systems Committee and the four subcommittees (ISO 17025, Microbiology, Asbestos, and PBMS) for their hard work and dedication this past year. Dr. Siegelman reviewed the voting process, complete details of which are shown in Attachment C.

ISO 17025 DRAFT

As Chair of the ISO 17025 Subcommittee, Dr. Siegelman reviewed the background of the International Organization for Standardization (ISO) 17025, which was approved in 1999 and replaced ISO/IEC Guide 25. The current NELAC Standard is organized to follow ISO Guide 25, which is becoming obsolete. Therefore, in order to remain consistent with the International ISO Standards, it is necessary to reorganize the NELAC Standard to match ISO 17025. Some new requirements, consistent with the 9000 series of standards, are: identification of potential conflict of interests, service to clients, preventive action, and uncertainty procedures. The new ISO 17025-based Standard will be cross-referenced with the current NELAC Standard in Appendix F. Complete details of Dr. Siegelman's presentation are shown in Attachment D.

It was suggested that this ISO 17025-based Standard be adopted in 2005; however, it was agreed that this was not an issue relevant to this Committee. Therefore, if voted in, the ISO 17025-based Standard would be effective according to the current policy of a two-year implementation cycle.

Marlene Moore noted that Chapter 5 contains two separate definitions for the term "procedure", resulting in a conflict with Accrediting Authorities. The Program Policy and Structure Committee has agreed to add a definition of "procedure" to the glossary in Chapter 1 during this Conference. The source of that definition is from the current ISO 9000/2000 Standard. Ms. Moore also noted that ISO 17025 will require less documentation than ISO 25 has. She requested that the Quality Systems Committee review the terms "procedure", "protocol", "Standard Operating Procedures", and "test methods" in the language of Chapter 5 as an action item for the next Conference.

An attendee raised an issue regarding some confusion with the language found in section 5.5.6.2.1 that deals with Calibration Laboratories. Bob Di Rienzo clarified that if a laboratory does not issue a calibration certificate, it is not obligated to follow the requirements for calibration laboratory. The ISO Subcommittee will continue to deliberate on this language in future teleconferences.

Dr. Siegelman invited the attendees to submit proposed text on any area of Chapter 5 to the Quality Systems Committee at any time for the members' future consideration and revisions.

The previously submitted changes to ISO 17025 were accepted as proposed with no further modifications.

ASBESTOS

Mike Beard, Co-chair of the Asbestos Subcommittee, reviewed the proposed changes to the Asbestos portion of Appendix D via speakerphone. Mr. Beard noted that in section D.6.6.1.2, Air, an error reading "mm^2" will be changed to "cc".

Attendees had concerns regarding the use of "hazardous waste" in the titles that are found throughout the appendix. After some discussion, it was agreed that a global change of the title **"*Solid and Hazardous Waste (Bulk)"*** to "**Bulk Samples"** will be made to the appendix.

It was suggested that after NELAC 8, the Asbestos Subcommittee review the proposed language that deals with "positive controls" for further clarification.

DATA INTEGRITY

Mr. Di Rienzo reviewed the proposed changes to data integrity. Although possible modifications to the proposed changes were discussed, none was recommended to be presented to the voting members. His presentation is shown in Attachment E.

MICROBIOLOGY

Marty Casstevens introduced the members of the Microbiology Subcommittee and reviewed the changes proposed to the microbiology sections of Chapter 5. The changes proposed are as follows:

CULTURE MEDIA
No comments were made nor changes proposed to this section.

TESTING MICROBIOLOGY SAMPLES FOR FREE CHLORINE

Section 5.11.3 Sample Receipt Protocols (applies also to "NELAC Plus 17025" 5.5.8.3.1.a.2)

2) The laboratory shall implement procedures for checking chemical preservation using readily available techniques, such as pH or ~~*free*~~ chlorine, prior to or during sample preparation or analysis.

Microbiological samples from *chlorinated* ~~public water~~ systems do not require an additional chlorine residual check in the laboratory if the following conditions are met:

i. sufficient sodium thiosulfate is added to each container to neutralize at minimum 5 mg/l of chlorine *for drinking water* ~~*samples*~~ *and 15mg/l of chlorine for wastewater samples;*

ii. one container from each batch of laboratory prepared containers or lot of purchased ready-to-use containers is checked to ensure efficacy of the sodium thiosulfate to 5 mg/l chlorine ~~and the check is documented *to single chlorine*~~ *or 15mg/l chlorine as appropriate and the check is documented;*

iii. chlorine residual is checked in the field and ~~*documented on the chain of custody*~~ *and actual concentration is documented with sample submission.*

FILTRATION SERIES, SANITIZING FUNNELS

D.3.1 Sterility Checks and Blanks, Positive and Negative Controls

a2) For ~~each filtration series in the~~ filtration technique, the laboratory shall ~~prepare at least~~ conduct one beginning and one ending sterility check for each *laboratory sterilized* filtration unit used in a filtration series. The filtration series may include single or multiple filtration units, which have been sterilized prior to beginning the series. *For pre-sterilized single use funnels a sterility check shall be performed on one funnel per lot.* ~~When an interruption of more than 30 minutes occurs, the filtration funnels shall be re-sterilized.~~ The filtration series is considered ended when more than 30 minutes elapses between successive filtrations. During a filtration series, filter funnels must be rinsed with three 20-30 ml portions of sterile rinse water after each sample filtration. In addition, laboratories must insert a sterility blank after every 10 samples or sanitize filtration units by UV light after each sample filtration.

CHEMISTRY

Charles Hooper presented the proposed changes to the chemistry section of Chapter 5. After some discussion, the following modifications were made to the earlier changes, to be presented for voting:

ONE STANDARD CALIBRATION, QUANTITATION AND CONTINUING CALIBRATION, DATA QUALIFIERS FOR INITIAL CALIBRATION, AND TWO STANDARDS AND A BLANK

Section 5.9.4.2 Instrument Calibration

~~**Note: In the following sections, initial instrument calibration is directly used for quantitation and continuing instrument calibration verification is used to confirm the continued validity of the initial calibration.**~~ **Note: In the following sections, initial instrument calibration is directly used for quantitation and continuing instrument calibration verification is used to confirm the continued validity of the initial calibration**~~–~~***unless otherwise required by regulation, method, or program.***

~~e~~c~~)~~ ~~Sample results must be quantitated from the initial instrument calibration and may not be quantitated from any continuing instrument calibration verification.~~ Sample results must be quantitated from the initial instrument calibration and may not be quantitated from any continuing instrument calibration *verification* ***unless otherwise required by regulation, method, or program.***

~~f~~ef) Results of samples ~~*not bracketed by initial instrument calibration standards (within calibration range)*~~ *outside of the concentration range established by the initial calibration* must be reported~~ as having less certainty, e.g.,~~ with defined qualifiers or flags or explained in the case narrative. The lowest calibration standard must be above the detection limit. Noted exception: The following shall occur for instrument technology (such as ICP or ICP/MS) with validated techniques from manufacturers or methods employing standardization with a zero point and a single point calibration standard: ~~*Standardization of the instruments using the zero point and single standard shall be performed with each analytical batch. Once the instrument is standardized with the zero point and the single standard, the linear working range must then be defined by the analysis of a series of reference standards, one of which must be at the minimum quantitation limit. Once the linear range is established it shall be routinely checked at a frequency and using procedures as established by the method and/or manufacturer. The minimum quantitation limit (MQL) shall be demonstrated with each analytical batch by the analysis of a reference standard at a concentration corresponding to the MQL, with results meeting established acceptance criteria. If an individual sample analysis produces results above the single point calibration standard, one of the following actions shall occur: (1) analyze a reference standard at or above the sample value that meets established acceptance criteria for validating the linearity; (2) dilute the sample such that the result falls below the single point calibration concentration; (3) report the data with an appropriate data qualifier and/or explain in the case narrative.*~~

- *Prior to the analysis of samples the zero point and single point calibration must be analyzed and the linear range of the instrument must be established by analyzing a series of standards, one of which must be at the lowest quantitation level.*

- *Zero point and single point calibration standard must be analyzed with each analytical batch.*

- *A standard corresponding to the lowest quantitation level must be analyzed with each analytical batch.*

- *The linearity is verified at a frequency established by the method and/or the manufacturer.*

- *If a sample within an analytical batch produces results above its associated single point standard then one of the following should occur:*

1) analyze ~~a~~ reference ~~*standard*~~ *material* at or above the sample value that meets established acceptance criteria for validating the linearity;
2) dilute the sample such that the result falls below the single point calibration concentration;
3) report the data with an appropriate data qualifier and/or explain in the case narrative.

~~g~~fg) If the initial instrument calibration results are outside established acceptance criteria, corrective actions must be performed and all associated samples reanalyzed. If ~~*for any reason*~~ reanalysis of the samples is not possible, ~~D~~data associated with an unacceptable initial instrument calibration shall ~~*not*~~ be reported with~~*out*~~ appropriate data qualifiers.

~~i~~*h*i) If a reference or mandated method does not specify the number of calibration standards, the minimum number is two, *(one of which must be at the lowest quantitaion limit)* not including blanks or a zero standard with the noted exception of instrument technology for which it has been established by methodologies and procedures that a zero and a single point standard are appropriate for calibrations (see 5.9.4.2.1.f). The laboratory must have a standard operating procedure for determining the number of points for establishing the initial instrument calibration.

LCS

Betty Boros-Russo presented the proposed changes to the Laboratory Control Sample (LCS) via speakerphone. Many attendees expressed concerns and questions regarding the purpose of LCS. As a previous Quality Systems Committee Chair, Silky Labie reminded the attendees that the original intent of the LCS was to be used as a quality control measure over the entire batch. The previously proposed changes to Section D.1.1.b) were then accepted with no further modifications.

MS/MSD

Ms. Boros-Russo presented the proposed changes to the matrix spike/matrix spike duplicate (MS/MSD). After some discussion, the following modifications were made to the earlier changes and will be presented for voting:

D.1.1 Positive and Negative Controls

c) Sample Specific Controls

Evaluation Criteria and Corrective Action:	The results from matrix spike/matrix spike duplicate are primarily designed to assess the precision and accuracy of analytical results in a given matrix and are expressed as percent recovery (%R), ~~and~~ relative percent difference (RPD) or other appropriate statistical technique that allows comparison to established acceptance criteria. The laboratory shall document the calculation for %R, ~~*relative percent difference*~~ *RPD* or other statistical treatment used.

GENERAL: CLIENT NOTIFICATION

Dr. Siegelman reviewed the proposed changes to the language regarding client notification. The modifications to the proposed language are as follow:

5.4.4.1 The laboratory shall establish and maintain procedures for the review of requests, tenders and contracts. The policies and procedures for these reviews leading to a contract for environmental testing and/or calibration shall ensure that:

b) the laboratory has the capability and resources to meet the requirements;

The purpose of this review of capability ~~should~~ is to establish that the laboratory possesses the necessary physical, personnel and information resources, and that the laboratory's personnel have the skills and expertise necessary for the performance of the environmental tests and/or calibrations in question. The review ~~may~~ ~~*must*~~ *may* ~~also~~ encompass results of earlier participation in interlaboratory comparisons or proficiency testing and/or the running of trial environmental test or calibration programs using samples or items of known value in order to determine uncertainties of measurement, detection limits ~~of detection~~, confidence limits, ~~etc~~ or other essential quality control requirements. The current accreditation status of the laboratory must also be reviewed. The laboratory must inform the client of the results of this review if it indicates any potential conflict, deficiency, *lack of appropriate accredtation status*, or inability on the laboratory's part to complete the client's work. ~~*Suspension of accreditation, revocation of accreditation, or voluntary withdrawal of accreditation must be reported to the client.*~~

5.4.4.5 If a contract needs to be amended after work has commenced, the same contract review process shall be repeated and any amendments shall be communicated to all affected personnel. *Suspension of accreditation, revocation of accreditation, or voluntary withdrawal of accreditation must be reported to the client.*

GENERAL: SUBCONTRACTING

George Kulasingam reviewed the proposed changes to the language regarding subcontracting. The modifications to the proposed language are as follows:

5.4.5.4 The laboratory shall maintain a register of all subcontractors that it uses for environmental tests and/or calibrations and a record of the evidence of compliance with ~~*this Standard for the work in question*~~ *5.4.5.1*.

DISCUSSION: PBMS

Ken Jackson, member of the PBMS Subcommittee, presented a progress report of the Subcommittee's activities to date. The Subcommittee has been working with the comments and concerns that were voiced at NELAC 7i regarding the Standard that had been presented at that time. The Subcommittee has been working on reviewing the ISO 17025 language that pertains to method selection/modification/validation, as well as revising Appendix C to incorporate a tiered approach to method evaluation/validation. A draft document compiled by the Subcommittee, which is to be merged into Chapter 5, is shown in Attachment F. It is the goal of the Subcommittee to have it ready for discussion at NELAC 8i, and up for vote at NELAC 9. Dr. Jackson's full presentation is shown in Attachment G.

ADJOURNMENT

Dr. Siegelman thanked all members and attendees for their patience and perseverance during this meeting. There being no further business, the meeting was adjourned.

ATTACHMENT A

ACTION ITEMS
QUALITY SYSTEMS COMMITTEE MEETING
JUNE 10, 2002

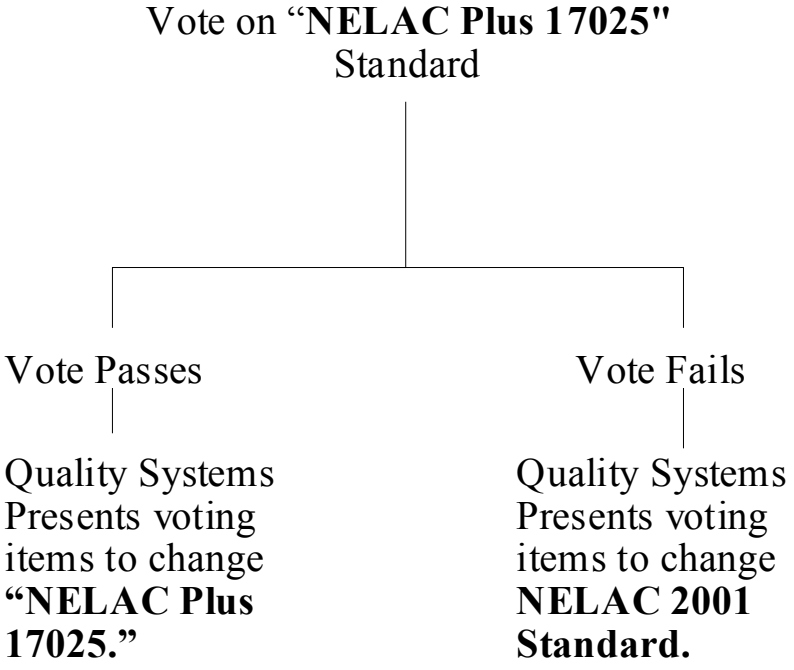
Item No.	Date Proposed	Action	Date to be Completed
1.	07/11/02	Committee to review the terms "procedure", "protocol", "Standard Operating Procedures", and "test methods" in the language of Chapter 5.	NELAC 9
2.	07/11/02	Asbestos Subcommittee will review the proposed language that deals with "positive controls" for further clarification.	Date to be determined

ATTACHMENT B

LIST OF PARTICIPANTS
QUALITY SYSTEMS COMMITTEE MEETING
JULY 10, 2002

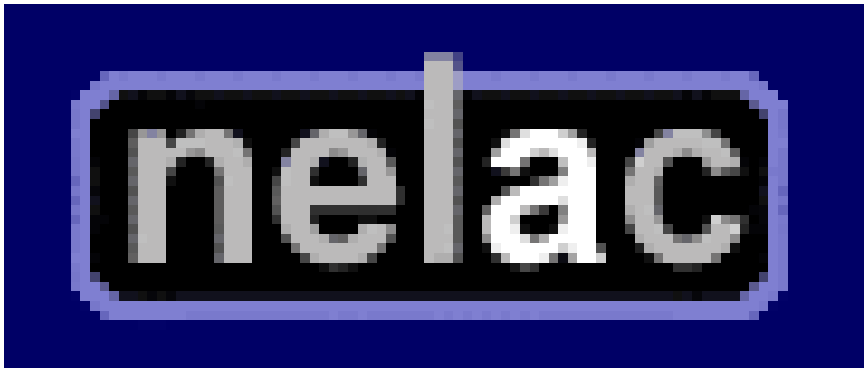
Name	Affiliation	Address
Frederic Siegelman Chairperson	USEPA/OEI	T: (202) 564-5173 F: (202) 565-2441 E: siegelman.frederic@epa.gov
Betty Boros-Russo (Absent)	New Jersey Dept. of Environmental Protection-OQA	T: (609) 292-3950 F: (609) 777-1774 E: bboros@dep.state.nj.us
Marty Casstevens	Lancaster Laboratories	T: F: E: mcasstevens@lancasterlabs.com
Peter De Lisle (Absent)	Coastal Bioanalysts, Inc.	T: (804) 695-8285 F: (804) 695-1129 pdelisle@coastalbio.com
Bob Di Rienzo	DataChem Laboratories	T: (801) 266-7700 F: (801) 268-9992 E: dirienzo@datachem.com
Clifford Glowacki	Technicon, LLC	T: (916) 929-8001 F: (916) 929-8020 E: cglowacki@technikonllc.com
Charles Hooper	USEPA/Region 4	T: (706) 355-8838 F: (706) 355-8803 E: Hooper.Charles@epa.gov
Kulasingam, George	CA State, Dept. of Health Services – ELAP	T: (510) 540-2800 F: (510) 849-5106 E: gkulasin@dhs.ca.gov
David Mendenhall (Absent)	Utah Department of Health	T: (801) 584-8470 F: (801) 584-8501 E: davidmendenhall@utah.gov
Jeffrey Nielsen	City of Tallahassee, Water	T: (850) 891-1232 F: (850)-891-1062 E: nielsenj@mail.ci.tlh.fl.us
Gabrielle Porath (Contractor Support)	Anteon Corporation	T: (702) 731-4158 F: (702) 731-4027 E: gporath@anteon.com

ATTACHMENT C



ATTACHMENT D

ISO 17025 PRESENTATION
QUALITY SYSTEMS COMMITTEE MEETING
JUNE 10, 2002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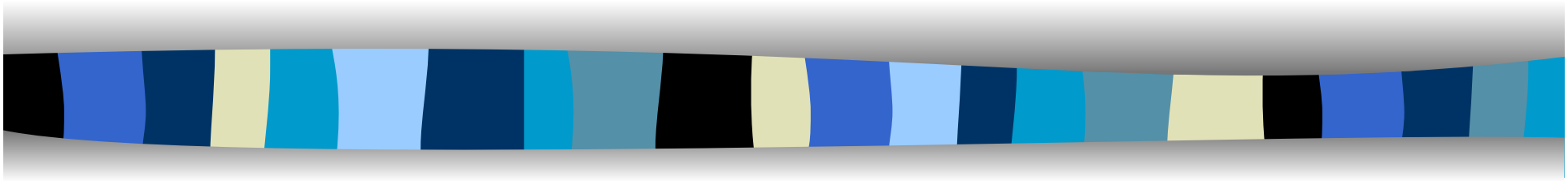
NELAC Quality Systems
NELAC VIII Annual Meeting
July, 2002



Fred Siegelman
Quality Staff
Office of Environmental Information
U.S. Environmental Protection Agency
Washington, DC 20460

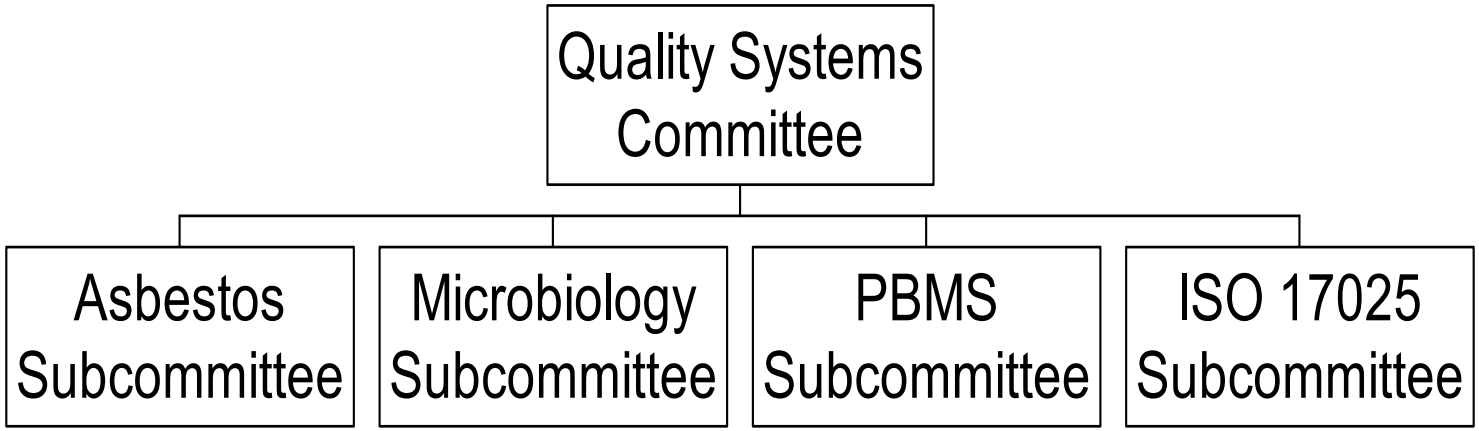
NELAC Quality System Goals

- Improve overall quality of compliance data via NELAC/NELAP
- Improve present NELAC Quality Systems standards
- Utilize ISO/IEC 17025 standard
- Further utilize PBMS concepts
- Adhere to Quality Systems Committee's Guiding Principles

AGENDA

9:00 AM - 9:15 AM	**Welcome & Introductions**
9:15 AM - 10:15 AM	**ISO 17025 Draft**
10:15 AM - 10:30 AM	**Asbestos**
10:30 AM - 11:00 AM	BREAK
11:00 AM - 11:15 AM	**Asbestos**
11:15 AM - 11:45 AM	**Data Integrity**
11:45 AM - 12:00 PM	**Microbiology**: Culture Media
12:00 PM - 12:15 PM	**Microbiology**: Testing Samples for free chlorine
12:15 PM - 12:30 PM	**Microbiology**: Filtration series, Sanitizing Funnels
12:00 PM - 1:30 PM	LUNCH
1:30 PM - 1:50 PM	**Chemistry**: One standard calibration
1:50 PM - 2:10PM	**Chemistry**: Quantitation and Continuing Calibration
2:10 PM - 2:30 PM	**Chemistry**: Data Qualifiers for Initial Cal.
2:30 PM - 2:50 PM	**Chemistry**: Two standards and a blank
2:50 PM - 3:00 PM	**Chemistry**: LCS
3:00 PM - 3:30 PM	BREAK
3:30 PM - 3:50 PM	**Chemistry**: MS/MSD
3:50 PM - 4:0O PM	**General**: Client Notification
4:00 PM - 4:15 PM	**General**: Subcontracting
4:15 PM - 4:45 PM	**PBMS:** Discussion
4:45 PM - 5:00 PM	**Closing**

NELAC Quality Systems



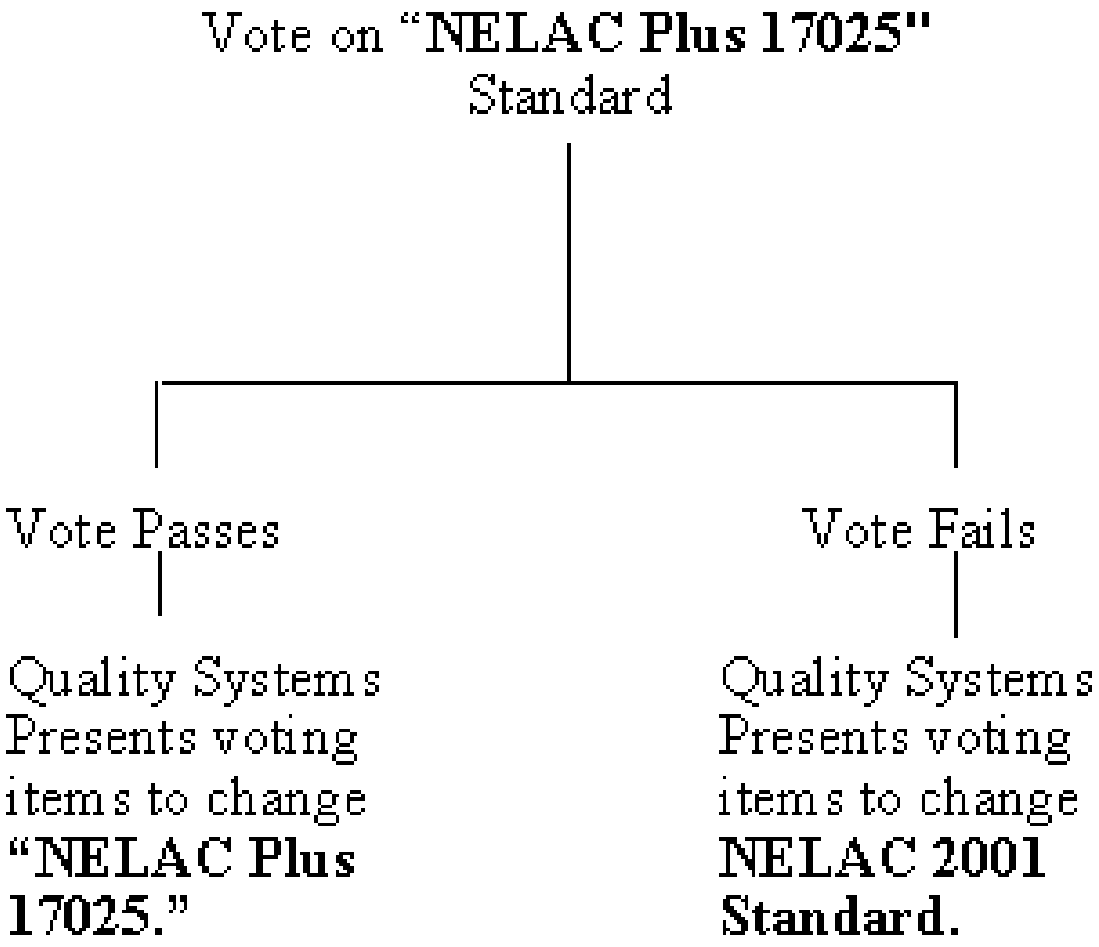
Quality Systems Committee

Ms. Betty J. Boros-Russo, New Jersey
Ms. Martha Casstevens, Lancaster Labs
Dr. Peter F. De Lisle, Coastal Bioanalysts, Inc.
Mr. Robert P. Di Rienzo, DataChem Laboratories
Mr. Clifford R. Glowacki, TECHNIKON, LLC
Mr. Charles H. Hooper, USEPA
Dr. George Kulasingam, California
Mr. David Mendenhall, Utah
Mr. Jeffrey Nielsen, City of Tallahassee
Dr. Frederic L. Siegelman, USEPA

NELAC Quality System Activities

- ISO 17025 based draft standard
- Asbestos
- Data Integrity
- Microbiology
- Performance Based Measurement Systems (PBMS)

Voting Process



Supporting Documents

- *NELAC 8 Quality Systems Voting Items Explanation*
- *NELAC Plus 17025*
- *ISO 17025 Abstract of Proposed Changes*
- *Quality Systems Abstract of Proposed Changes*
- *NELAC 8 Proposed Changes to ISO 17025: Quality System*.
- *NELAC 8 Proposed Changes to Chapter 5: Quality System*

More Information

- NELAC home page:
  - –http://www.epa.gov/ttn/nelac
- Quality Staff home page:
  - –http://www.epa.gov/QUALITY/

Agenda

- ISO 17025 Draft
- Asbestos
- Data Integrity
- Microbiology
- Chemistry
- General
- PBMS Discussion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ISO/IEC 17025: General Requirements for the Competence of Testing and Calibration Laboratories

- Approved in 1999
- Replaced third edition of ISO/IEC Guide 25:1990

NELAC and ISO 17025 Issues

- NELAC Quality Systems present standard based on ISO Guide 25
- ISO/IEC 17025 replaced ISO Guide 25
- ISO/IEC 17025 - ANSI Copyright Issue

ISO 17025 Based Version of the NELAC Standard Alternatives

- NELAC standard consistent with the international standard ISO 17025
- NELAC standard organized to follow ISO 17025
- NELAC standard includes the ISO 17025 Language

Management Requirements

- 5.4.1 Organization
- 5.4.2 Quality System
- 5.4.3 Document Control
- 5.4.4 Review of Requests, Tenders, & Contracts
- 5.4.5 Subcontracting of Environmental Tests & Calibrations

Technical Requirements

- 5.5.1 General
- 5.5.2 Personnel
- 5.5.3 Accommodation & Environmental Conditions
- 5.5.4 Environmental Test & Calibration Methods and Method Validation
- 5.5.5 Equipment

ISO 17025 Subcommittee

Debra Conner, USEPA
Skip Darley, Navy
Bob Di Rienzo, Datachem
John Gumpper, Chemval
Deb Henderer, Paragonlabs
Carl Kircher, Florida
Don Lore, Utah
Barbara McCleary, Delaware
Marlene Moore, Advanced Systems
Randy Querry, A2LA
Don Zahniser, Kodak
Betsy Ziomek, Virginia
Fred Siegelman, USEPA

ISO 17025 New Requirements

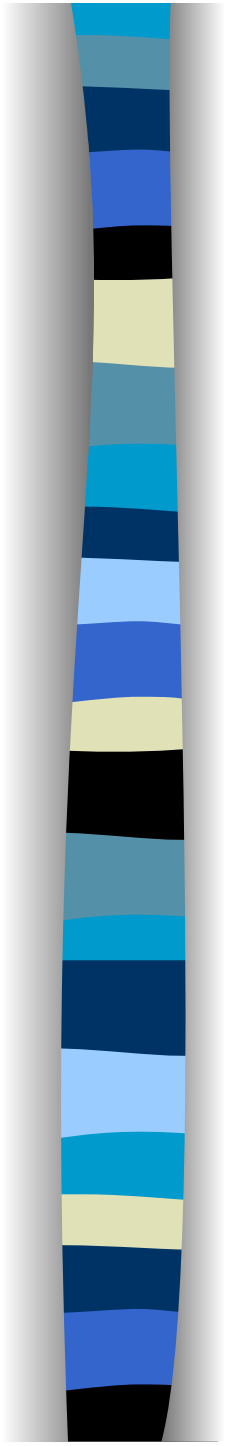
- Consistent with ISO 9000 series standards
- Identification of potential conflicts of interest
- Service to clients
- Preventive action
- Uncertainty procedures for testing

Proposed Organization for NELAC Chapter 5

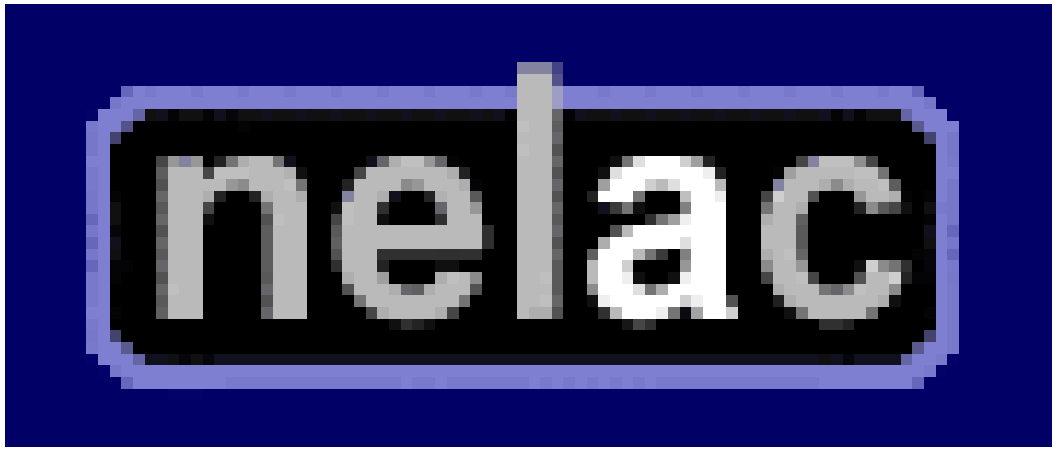
- 5.0 Quality Systems - Introduction
- 5.1 Scope
- 5.2 References
- 5.3 Terms & Definitions
- 5.4 Management Requirements
- 5.5 Technical Requirements

APPENDIX F
CROSS-REFERENCE TO NELAC 2001

NELAC 2001 Chapter 5	NELAC 2001 text begins with:	NELAC 2002 Chapter 5 (ISO 17025 Format)
5	*QUALITY SYSTEMS*	5
5.1	*SCOPE*	5.1
5.1.a	*This Standard sets out the general requirements that a laboratory has*	5.1.1
5.1.b	*This Standard includes additional requirements and information for*	5.1.1 and 5.1.4
5.1.c	*This Standard is for use by environmental testing laboratories in the*	5.1.4
5.2	*REFERENCES*	5.2
5.3	*DEFINITIONS*	5.3
5.4	*ORGANIZATION AND MANAGEMENT*	5.4
5.4.1	*Legal Definition of Laboratory*	5.4.1.1 and 5.4.1.2
5.4.2	*Organization*	5.4.1.5
5.4.2.a	*have managerial staff with the authority and resources needed to*	5.4.1.5.a



Questions?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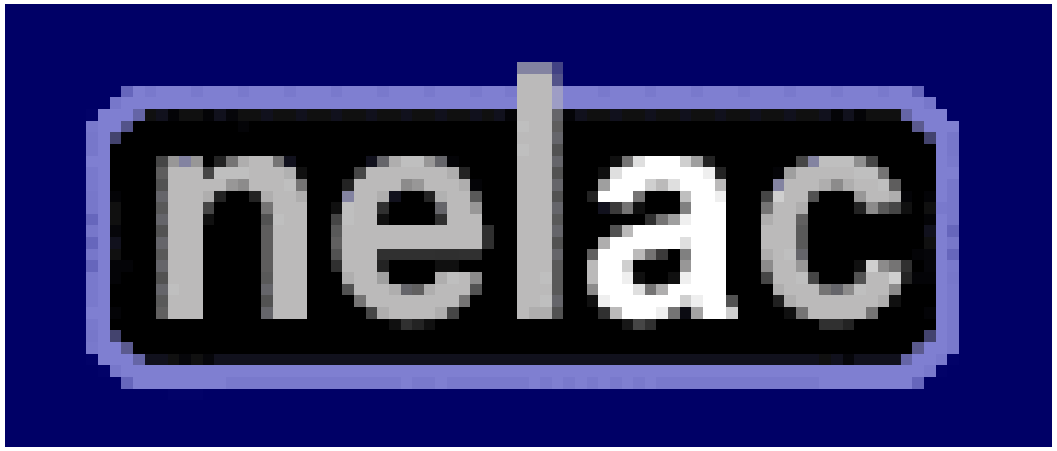
General: Client Notification

- Change to the NELAC plus 17025 draft only.
- Review of capabilities in response to a request is a requirement.
- Review must include results of proficiency testing
- Review must include review of Accreditation status
- Client must be informed of results of review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ATTACHMENT E

DATA INTEGRITY
QUALITY SYSTEMS COMMITTEE MEETING
JUNE 10, 2002

Data Integrity
NELAC 2001
Chapter 5

NELAC 2001 – "Quality Manual"
Section 5.5.2.u

Ethics Policy Statement

Training for Employees

Legality of Improper Behavior

Data Integrity
NELAC 2001
Chapter 5

NELAC 2001 “Internal Audits
Section 5.5.3.1

Internal Audits to verify compliance with Quality System

Data Integrity
NELAC 2001
Chapter 5

NELAC 2001 “Laboratory Management Responsibilities
Section 5.6.2.h

Developing a proactive program for
prevention and detection of improper,
unethical or illegal actions….

Data Integrity Procedures

Two versions of Data Integrity Procedures are presented as additions/revisions to Chapter 5 Quality Systems

- ✓ Version 1 – NELAC 2001 plus ISO 17025
- ✓ Version 2 – NELAC 2001 Chapter 5

Summary of Data Integrity Procedures

The Four Elements of Data Integrity

- Employee Training
- Employee Training Documentation
- Data Integrity Reviews and Procedures
- Data Integrity Procedures Documentation

NELAC 2001 plus ISO 17025

- **Employee Training**

  Provided for all employees
  See Section 5.5.2.7

- **Employee Training Documentation**

  Documentation that all staff has participated in and understands their obligations related to Data Integrity
  See Section 5.5.2.7

NELAC 2001 plus ISO 17025

➢Data Integrity Reviews and Procedures

A confidential mechanism for employees to report data integrity issues.
See Section 5.4.2.6.1

Internal Audits shall be conducted with respect to any evidence of inappropriate actions or vulnerabilities related to data integrity.
See Section 5.4.15

NELAC 2001 plus ISO 17025

➢Data Integrity Procedures Documentation

Documentation of Investigations and Internal Audits
See Section 5.4.15

Data Integrity Procedures reviewed annually
See Section 5.4.2.6

How the NELAC 2001 plus ISO 17025 ➢Draft Changes?

Delete Sections:
5.4.2.3.u
5.5.2.6.c.3
5.5.2.6.h

Add New Sections:
5.1.7
5.4.2.6, 5.4.2.6.1, and 5.4.2.6.2
5.5.2.7
5.4.15

➢ How the NELAC 2001 Chapter 5 Standard Changes?

Add New Sections:
5.1.d
5.5.3.1.1
5.5.3.6

Replace Existing Sections:
5.5.2.u
5.5.3
5.6.2.h

QUESTIONS

NELAC 8

Quality Systems Committee
PBMS Subcommittee's

DRAFT DISCUSSION DOCUMENT (i.e., work-in-progress)

July 7 – July 12, 2002

Tampa, Florida

5.0 Analytical methods

Each laboratory shall have a quality system. The laboratory's Quality System is the process by which the laboratory conducts its activities so as to provide the client with data of known and documented quality with which to demonstrate regulatory compliance and for other decision-making purposes. This system includes a process by which appropriate analytical methods are selected, their capability is evaluated, and their performance is documented. The quality system shall be documented in the laboratory's quality manual.

This chapter contains detailed quality system requirements for consistent and uniform implementation by both the laboratories conducting testing under these standards and the evaluation of those laboratories by accrediting authorities. Each laboratory seeking accreditation under NELAP must assure that they are implementing their quality manual and that all the Quality Control (QC) procedures specified in this Chapter are being followed. The Quality Assurance (QA) policies, which establish essential QC procedures, are applicable to environmental laboratories regardless of size and complexity.

All items identified in this Chapter shall be available for on-site assessment or data audit

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5.5.4.1 Standard Operating Procedures and Laboratory Manual(s)

The laboratory shall maintain a methods manual consisting, at a minimum, of all the laboratory's standard operating procedures (SOP's). The SOP's shall accurately reflect all phases of current laboratory activities such as sample receipt, sample storage, sample analysis, assessment of data integrity, corrective actions, handling of customer complaints, test methods, and data and record storage. All confidential business information in the methods manual shall be so designated and appropriately marked by the laboratory.

a) An SOP may be an equipment manual provided by a manufacturer, or an internally written document so long as the SOP is adequately detailed to permit someone other than the analyst to reproduce the procedures that had been used to produce a given result.

b) The test method SOP's may be copies of published methods as long as any changes or selected options in the methods are documented and included in the SOP's (see below). Reference test methods that contain sufficient and concise information on how to perform the tests do not need to be supplemented or rewritten as internal procedures if these methods are written in a way that they can be used as published by the laboratory. It may be necessary to provide additional documentation for optional steps in the method or additional details.

c) Copies of all SOP's shall be accessible to all appropriate personnel.

d) SOP's shall be organized in a manner such that they are easily accessible to the laboratory staff.

e) Each SOP shall clearly indicate its effective date, its revision identifier, and shall bear the signature(s) of the approving authority.

f) Each test method SOP shall give or reference the following information, where applicable (the order in which these items appear in the SOP is left to the discretion of the laboratory staff):

1.0 Scope and Application
2.0 Summary of Method
3.0 Definitions

4.0 Interferences
5.0 Safety
6.0 Equipment and Supplies
7.0 Reagents and Standards
8.0 Sample Collection, Preservation, and Storage
9.0 Quality Control
10.0 Calibration and Standardization
11.0 Procedure
12.0 Data Analysis and Calculations
13.0 Method Performance
14.0 Pollution Prevention
15.0 Waste Management
16.0 References
17.0 Tables, Diagrams, Flowcharts, and Validation Data

5.5.4.2 Selection of Methods

The laboratory shall utilize methods within its scope (including sample collection, sample handling, transport and storage, sample preparation and sample analysis) that are appropriate and applicable to client needs (i.e., to meet regulatory or other requirements specified by the client). These requirements may specify that a particular method or group of methods be employed for a given project or program, or that specific measurement quality objectives be achieved, or both.

When the use of a particular test method is mandated by regulation or requested by a client, only that method shall be used. Deviations from a test method shall occur only if the deviation has been documented, technically justified, authorized, and approved for use by the client. The laboratory shall inform the client when the method proposed by the client is considered not capable of providing data consistent with intended use. Client approval of the methods to be used when conducting analyses must be obtained prior to implementation. Modifications must be documented in communication with and/or reports to the client.

When the use of a particular test method is not either mandated by regulation or requested by a client, the laboratory shall select methods that are appropriate for the intended use. Such methods may be those published in international, regional, or national standards, or by reputable technical organizations, or in relevant scientific texts or journals, or as specified by the manufacturer of the equipment, or laboratory-developed methods or methods adapted by the laboratory. The laboratory shall document in reports to its clients all methods utilized in the performance of work.

5.5.4.3 Method Evaluation and Performance Demonstration

All measurements made while operating as a NELAC accredited laboratory must have an adequate demonstration that the measurement system provided data consistent with its intended use. This demonstration consists of three activities:

1) an initial evaluation that the measurement system is capable of providing data of the quality needed to meet client and/or regulatory requirements;

2) an acceptable instrument calibration and verification that the system has remained calibrated during the period that it was used for analysis ; and

3) an on-going demonstration of measurement system performance that documents the laboratory is operating with its analytical system in control as well as a documentation of the quality of data obtained on the actual samples analyzed.

5.5.4.3.1 Initial Measurement System Evaluation

Each laboratory must evaluate the capability of its measurement system relative to its intended purpose. The thoroughness and robustness of the evaluation depends on what is already known about the performance of the method on the analyte-matrix combination of concern over the concentration range of interest as well as the intended use of the data. Properties of the measurement system to be evaluated include bias, precision, sensitivity, and selectivity. The measurement system includes the analyst (operator) or work cell and method.

Essential elements of the measurement system evaluation include determination of accuracy (i.e., bias, and precision), confirmation of adequate sensitivity, determination of the range of measurement capability, and verification of adequate system selectivity for the intended purpose.

Procedures for the initial measurement system evaluation are presented in Appendix C. The laboratory shall record the results of the evaluation (measurement quality characteristics, MQC), the protocol used for the evaluation, and the measurement performance (measurement quality objectives, MQO). When changes are made in a method, the influence of such changes shall be documented and, if appropriate, a new evaluation shall be carried out.

5.5.4.3.2 On-going Demonstration of Measurement System Performance

In addition to the requirement for an initial evaluation, the following general quality control (QC) procedures, used to demonstrate that the laboratory analytical system was functioning correctly and to document the performance of the method when used to analyze samples, shall apply. The manner in which they are implemented is dependent on the types of tests performed by the laboratory (i.e., chemical, whole effluent toxicity, microbiological, radiological, air) and is further described in Appendix D.

a) The laboratory shall have QC procedures in place to monitor the performance of the measurement system on an on-going basis, including:

(1) procedures to verify that the instrument is calibrated;

(2) procedures to ensure that the measurement system is free of laboratory induced interferences;

(3) procedures to identify if and when the laboratory is in an out-of-control condition;

(4) procedures to document the sensitivity, precision and bias of the results for the samples analyzed;

(5) procedures to confirm analyte identity and quantitative accuracy; and

(6) procedures to verify continuing analyst proficiency.

b) All quality control measures shall be assessed and evaluated on an on-going basis, and quality control acceptance criteria shall be used to determine the usability of the data. (See Appendix D.) The essential quality control measures for chemical testing are found in Appendix D.1 of this Chapter.

c) The laboratory shall have procedures for the development of acceptance criteria and associated corrective action procedures for all QC activities where no analogous method or regulatory criteria or procedures exist.

d) To the extent possible, samples shall be reported only if all quality control measures are acceptable. If a quality control measure is found to be out of control and the data is to be reported, all samples associated with the failed quality control measure shall be reported appropriately.

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5.5.4.3.3 Calibration

Calibration requirements are divided into two parts: (1) requirements for analytical support equipment, and 2) requirements for instrument calibration. In addition, the requirements for instrument calibration are divided into initial instrument calibration and continuing instrument calibration verification.

5.5.4.3.3.1 Support Equipment

These standards apply to all devices that may not be the actual test instrument but are necessary to support laboratory operations. These include but are not limited to: balances, ovens, refrigerators, freezers, incubators, water baths, temperature measuring devices (including thermometers and thermistors), thermal/pressure sample preparation devices, and volumetric dispensing devices (such as Eppendorf or automatic dilutor/dispensing devices) if quantitative results are dependent on their accuracy, as in standard preparation and dispensing or dilution into a specified volume.

(a) All support equipment shall be maintained in proper working order. The records of all repair and maintenance activities including service calls, shall be kept.

(b) All support equipment shall be calibrated or verified at least annually, using NIST traceable references when available, over the range of use. The results of such calibration shall be properly documented and recorded, and shall be within the specifications required of the application for which this equipment is used or:

(1) The equipment shall be removed from service until repaired; or

(2) The laboratory shall maintain records of established correction factors to correct all measurements.

(c) Raw data records shall be retained to document equipment performance.

(d) Prior to use on each working day, balances, ovens, refrigerators, freezers, and water baths shall be checked in the expected use range, with NIST traceable references where available. The acceptability for use or continued use shall be according to the needs of the analysis or application for which the equipment is being used.

(e) Mechanical volumetric dispensing devices including burettes (except Class A glassware) shall be checked for accuracy on at least a quarterly use basis. Glass microliter syringes are to be considered in the same manner as Class A glassware but must come with a certificate attesting to established accuracy, or the accuracy must be initially demonstrated and documented by the laboratory.

(f) For chemical tests the temperature, cycle time, and pressure of each run of autoclaves must be documented by the use of appropriate chemical indicators or temperature recorders and pressure gauges.

(g) For biological tests that employ autoclave sterilization, see Section D.3.8.

5.5.4.3.3.2 Instrument Calibration

This standard defines the requirements that laboratories must follow to ensure that all instruments used for analysis are properly calibrated before and during their use in order for the data to be of known quality and appropriate for the intended use. This standard does not specify detailed procedural steps ("how to") for calibration but establishes the minimum essential elements. This approach allows flexibility and permits the employment of a wide variety of analytical procedures and statistical approaches. At a minimum these essential elements must be addressed during spectrochemical, electrochemical, and chromatographic test procedures. Other sections in this chapter address the essential elements for additional test procedures, such as Appendix D.4.4 for radiochemistry. If more stringent standards or requirements are included in the particular test method being used for analysis, the method standards shall apply and the laboratory demonstrate that such standards are met. If it is not apparent which standard is more stringent, then the requirements of the method or regulation are to be followed.

Note: In the following sections, initial instrument calibration (calibration) is directly used for quantitation and calibration verification is used to confirm the continued validity of the initial calibration.

5.5.4.3.3.2.1 Initial Calibration

The following items are essential elements of calibration:

a) All instruments shall be calibrated before use and maintained in a calibrated state during use.

b) The calibration shall be verified with a standard(s) prepared independent of the standards used for calibration. This verification shall be performed with a standard obtained from a second manufacturer, or it can be a second standard obtained from the manufacturer if the second lot can be demonstrated to have been prepared independently from the first lot.

c) The details of the calibration procedures including calculations, integrations, acceptance criteria and associated statistics shall be included or referenced in the test method SOP. When calibration procedures have been specified in a test method, then a copy of the method must be retained by the laboratory and be available for review. When the calibration procedure is specified by regulation or by the client (including the number of calibration points), calibration shall be performed as specified.

d) Results of the calibration must be documented and retained by the laboratory and be available for review. Sufficient raw data records must be retained to permit reconstruction of the calibration (e.g., calibration date, test method SOP, instrument identifier, each analyte name, analyst's name; concentrations used, and instrument responses obtained, calibration line or curve or response factor, or unique equation or coefficient used to convert analytical system responses to concentrations or amounts).

e) All sample results shall be quantitated from the calibration and shall not be quantitated from any calibration verification, unless specifically required by the method or client.

f) Calibrations shall be traceable to a national standard, when available. The lower calibration standard shall be at or below the limit of quantitation and the upper calibration standard at the highest

concentration that quantitative data are to be reported (see Appendix C). These two calibration standards define the working range of the calibration.

g) Measured concentrations that are outside of the working range shall be reported as having less certainty (e.g., defined qualifiers or flags or explained in the case narrative). The lowest demonstrated quantitation limit is the lowest concentration that data shall be reported with certainty.

h) If the calibration results are outside established acceptance criteria, corrective actions must be performed. Data associated with an unacceptable initial instrument calibration shall not be reported without qualifiers and explanation.

i) Criteria for the acceptance of calibration shall be established (e.g., correlation coefficient or relative standard deviation of calibration or response factors). The criteria used must be appropriate to the calibration technique employed.

j) If the method being employed for the analysis does not specify the number of calibration points, the minimum number must reflect the objectives of the analysis and the linearity of the instrument. For example, if the data only needs to show that a result is above or below a certain number (e.g., a regulatory limit), a single point calibration at that limit is sufficient. A single point standard at the reporting limit is also sufficient to demonstrate absence of the analyte. For detectors with a very linear response, a calibration line between a single point and a blank or zero may be sufficient. In this case, the sensitivity, linearity, and accuracy must be demonstrated by quantitation of known standards at the low, mid, and high points of the calibration. The laboratory must have standard operating procedures that clearly specify the number of points for establishing the initial instrument calibration.

5.5.4.3.3.2.2 Calibration Verification

When the initial instrument calibration is not performed on the day of analysis, the validity of the instrument calibration shall be verified prior to sample analyses by a calibration verification with each analytical batch. Calibration shall be verified before conducting any analyses and at the end of each analytical batch. The following items are essential elements of calibration verification:

a) The details of the calibration verification procedure, calculations, and associated statistics must be included or referenced in the test method SOP.

b) Calibration shall be verified for each compound, element, or other discrete chemical species, except for mixtures such as Aroclor-1254, Total Petroleum Hydrocarbons, or Toxaphene where a representative chemical related substance or mixture can be used.

c) Instrument calibration verification must be performed:

(1) at the beginning and end of each analytical batch (however, if an internal standard is used, only one verification needs be performed at the beginning of the analytical batch),

(2) whenever it is suspected that the analytical system may be out of calibration or might not meet the verification acceptance criteria,

(3) if the time period for calibration or the most previous calibration verification has expired, or

(4) for analytical systems that contain a calibration verification requirement based on the number of runs, the number of runs is exceeded.

d) Results of the calibration verification must be documented and retained by the laboratory and be available for review. Sufficient raw data records shall be retained to permit reconstruction of the calibration verification (e.g., verification date and time, test method SOP used, instrument identifier, each analyte name, analyst's name, concentrations used and instrument responses obtained, degree to which results matched calibration curve or response factor; and any equations or coefficients used to convert instrument responses to concentration). Calibration verification records must explicitly connect the verification data to the calibration (i.e. verification was performed on the same instrument and the most recent calibration was verified).

e) Criteria for the acceptance of a calibration verification must be established (e.g., relative percent difference from calibration).

f) If the calibration verification results are outside of acceptance criteria, corrective actions must be taken. Following completion of corrective actions, two immediately consecutive calibration verifications must be analyzed. If the two consecutive calibration verifications do not yield acceptable results, then the laboratory shall recalibrate the analytical system.

g) If the laboratory has not verified calibration, sample analyses shall not occur until the analytical system is calibrated or calibration is verified, with the exception that results associated with an unacceptable calibration verification may be reported as qualified data under the following special conditions:

(1) When the acceptance criteria for the calibration verification are exceeded high (i.e., high bias) and the analyte in the associated samples is not detected, then the non-detect may be reported. Otherwise, the samples affected by the unacceptable calibration verification shall be reanalyzed after the analytical system has been calibrated or calibration has been verified.

(2) When the acceptance criteria for the calibration verification are exceeded low (i.e., low bias) and the concentration or amount of the analyte in the associated samples exceeds a regulatory limit or decision level, the concentration or amount may be reported and appropriately qualified. Otherwise, the samples affected by the unacceptable verification shall be reanalyzed after the analytical system has been calibrated or calibration has been verified.

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5.6.2.4.c

4) Analyst training shall be considered up to date if an employee training file contains a certification that technical personnel have read, understood and agreed to perform the most recent version of the test method (the approved method or standard operating procedure as defined by the laboratory document control system, 5.5.2.d) and documentation of continued proficiency by at least one of the following once per year:

i. Acceptable performance of a blind PT sample (single blind to the analyst) Note: Successful analysis of a blind PT sample used for similar test methods using the same technology (e.g., GC/MS volatiles by purge and trap for Methods 524.2, 624 or 5035/8260) would only require documentation for one of the test methods;

ii. An initial measurement system evaluation as defined in Appendix C;

iii. At least four consecutive laboratory control samples with acceptable levels of precision and bias;

iv. If i-iii cannot be performed, analysis of authentic samples with results statistically indistinguishable from those obtained by another trained analyst.

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5.10.10 Documentation

For all environmental testing studies, the documentation of the results from the Initial Measurement System Evaluation, the instrument calibration and Ongoing Demonstration of Measurement System Performance (see Table A) shall be maintained with the laboratory records and reported to the client along with the actual test results when appropriate or requested.

Table 5-1

Evaluation Element	Initial Measurement System Evaluation	Ongoing Demonstration of Measurement System Performance
Calibration	Calibrate instrument	Calibrate instrument and/or verify calibration
Limit of Detection (LOD)	Establish LOD on each sample-type; if analytes are to be reported to LOD, analyze LOD QC sample,	Whenever analytes are to be quantitatively reported at LOD, analyze LOD QC sample with each sample batch.
Limit of Quantitation (LOQ)	Establish LOQ on each sample-type; verify by analysis of LOQ QC sample. Determine acceptance limits for LOQ QC samples	Analyze LOQ QC sample annually
Bias*	Establish acceptance limits for bias on each sample-type	Matrix spike/matrix spike duplicates for each batch per method requirements**
Precision*	Establish acceptance limits for precision on each sample-type	Matrix spike/matrix spike duplicates per method requirements, or replicate samples for each batch**
Method Range*	Establish working range	No requirement
Selectivity	Establish measures for ensuring selectivity criteria for each SOP	Confirm analyte identity and quantitative reliability for each positive result
Analytical System Performance	No requirement	Analyze Laboratory Control Sample with each batch
System Cleanliness	Analyze Method blank	Analyze Method blank
Analyst Proficiency	Initial demonstration of proficiency; see Appendix E	Periodic demonstration of proficiency; see Appendix E
Laboratory proficiency	Successful analysis of 2 PT samples; see Chapter 2	Annual analysis of 2 PT samples; see Chapter 2

* The specific requirements vary for standardized and non-standardized methods
** These QC samples are not required when the laboratory clients do not provide samples and/or MQOs.

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APPENDIX C TO NELAC STANDARDS CHAPTER 5
Initial Measurement System Evaluation Protocol

C.1 Purpose:

This Appendix serves to assess whether or not a particular measurement system is suitable for an intended purpose. This appendix also defines the documentation necessary to provide evidence that a measurement system was appropriately evaluated.). The laboratory shall ensure that the essential requirements in this Appendix are incorporated into their method manuals and/or the Laboratory Quality Assurance Plan.

The activities specified in this appendix are not suitable for demonstrating that a method, when considered independent of a laboratory's quality system, is valid. That activity generally requires a collaborative study such as is described in ASTM D-2777.

C.2 Background:

The bases for evaluating whether a measurement system's performance is suitable for a particular purpose are Measurement Quality Objectives (MQOs). The MQO elements are sensitivity, range, precision, bias, and selectivity. The measurement system is a method as implemented at a particular laboratory (i.e., the laboratory SOP, equipment and staff).

The range, bias, precision and sensitivity characteristics of each measurement system are determined by the laboratory using the procedures in Appendix C. These measures of system performance are defined as the measurement quality characteristics (MQCs). The selectivity of the measurement system is evaluated as part of the bias evaluation. If MQCs do not meet the respective MQOs, then the measurement system does not yield data suitable for its intended purpose

An Initial Measurement System Evaluation is done when the laboratory implements a method for the first time, significantly modifies a method that has previously been evaluated by the laboratory, adds an analyte to an existing method, or uses an existing method for a different sample-type. This evaluation is performed to demonstrate that the laboratory and method (the measurement system) is capable of providing data of the quality needed and to ensure data suitable for the intended purpose. The activities required for this evaluation are summarized in Table C-1.

When laboratory clients provide the laboratory with the required MQOs, these MQOs shall be used. When MQOs are not provided to the laboratory, the laboratory may use the performance characteristics of published standardized methods as MQOs, or may establish MQOs based on the MQC data obtained from an initial evaluation. If the measurement system performance is not adequate (i.e., does not meet one or more of the MQO requirements) for the intended purpose, the laboratory must notify the client prior to the analysis of client samples. If the method is modified to achieve improved MQOs, the initial evaluation must be repeated.

The role of the laboratory includes:

- Evaluate client MQOs and specific method requirements, if any, to assist in the method selection process.
- Perform the Initial Measurement System Evaluation to determine the associated Method Quality Characteristics (MQCs).
- Determine if the MQOs can be met and provide assurance of said performance to the client.
- Employ test methods that meet the needs of the client and ensure data suitable for its intended use.

C.3 Initial Measurement System Evaluation - General

The initial evaluation must be performed for all methods used at the laboratory, including:

- published standardized methods with no modifications;
- standardized methods that have been modified, and
- laboratory developed methods.

This evaluation (Section C.5) demonstrates the laboratory's ability to use the method correctly. Sample specific modifications (i.e., using a smaller sample size; adding a cleanup step) can be performed without re-doing the evaluation as long as the following conditions are met: the changes can be scientifically justified as not being ones that would change the nature of the procedure. The appropriate ongoing quality control sample analyses are used to document the measurement system performance; and both the changes and the rationale for the changes not having to be evaluated using the initial evaluation procedure are contained in the documentation (e.g., case narrative, corrective action form, non-conformance memo) of the analysis.

When a new analyte is added to an existing method, an initial evaluation must be performed for that analyte. Section C.5 details the steps involved in the initial evaluation. The bias and precision evaluation in Section C.5.2.2 must be followed for new analytes, unless the laboratory can demonstrate, by the nature of the analyte being added, that all measures of system performance can be assured (e.g., isomer of previously evaluated constituent that does not exhibit chromatographic interference with other target analytes) In the latter case, the bias and precision steps in Section C.5.2.1 must be followed. In other words, the initial evaluation must be sufficient to support the intended use of the data.

C.4 Matrices, Sample-Types and Quality Control Samples

An Initial Measurement System Evaluation must be performed for every method in the laboratory and for every sample-type to which the method is applied. The sample-types described below refer to a sample with certain properties within the broadly defined NELAC matrices, (Drinking Water; Non-Potable Water; Solid and Chemical Materials; Biological Tissues; and Air and Emissions) that provides a reasonable challenge to the method, but that does not address all potential matrix issues that could exist in actual samples. If the method is to be used on sample matrices which provide a more significant analytical challenge (e.g., sludges, chemical wastes, oils, brines), the on-going demonstration activities must be used to document the performance of the method in these other matrices. Alternatively, the laboratory may perform an initial evaluation on these sample matrices. Laboratories have the option to perform the initial evaluation on samples collected from a specific site (e.g., a POTW can use the wastewater discharged from their facility) provided the method is only used to analyze those samples or samples with comparable characteristics.

The sample-type for the Drinking Water matrix is tap water from the laboratory.

The Non-Potable Water sample type shall have the following characteristics:

Total suspended solids (TSS) greater than 40 mg/L
Total dissolved solids (TDS) greater than 100 mg/L
Soluble organic content greater than 20 mg/L
Salt content greater than 120 mg/L
Total Alkalinity greater than 140 mg/L

If the initial Measurement System Evaluation is performed on a Non-Potable Water sample-type, then the method may be applied to all Drinking Water and Non-Potable Water samples. However, if the initial evaluation is performed on a Drinking Water sample-type, the method may be applied to other sample-types within the Drinking Water matrix only.

For the Solid and Chemical Materials matrix the appropriate sample-type is a soil or sediment containing at least 10% each of sand, silt and clay and at least 5% moisture.

Within the Biological Tissues matrix the appropriate sample type is any fish or animal tissue that contains at least 5% fat.

Within the Air and Emissions matrix separate initial evaluations are required for canister or other whole-volume air samples, Polyurethane foam plugs (PUF) samples, filter media or the various absorption tube media.

The evaluation requires the laboratory to analyze various quality control (QC) samples. These samples may be:

- Certified Reference Materials (CRMs),
- The sample-types described above, fortified by spiking, or
- Actual samples, where the concentration of the analyte is known, either by an independent analysis or based on spiking.

C.5 MEASUREMENT SYSTEM EVALUATION

The table below summarizes each of the elements that must be evaluated and the requirements for performing the evaluation.

Table C-1. Summary of Initial Measurement System Evaluation Elements and Requirements*

Evaluation Element:	Evaluation Requirements
Limit of Detection	Determine LOD by EPA (40 CRF 136, App. B) or other established procedure for each analyte for each sample-type; include qualitative analyte identification and isolation/concentration steps, as appropriate. Determine for each instrument. Other options may be available, depending on agency/program requirements. If quantitative data are to be reported to the LOD, analyze a QC sample containing analytes at no more than 2X LOD.
Limit of Quantitation	Determine LOQ. Verification of calculated LOQ done by analysis of a QC sample containing

	analytes at LOQ. Calc. % recovery for each analyte.
Range	Establish the working range for each analyte as part of the calibration procedure.
Precision & Bias: standardized methods	Analyze QC sample containing analytes at 2-5X LOQ prior to any isolation/concentration steps; perform in quadruplicate. Verify & document reliable qualitative identification of analytes. Calculate % recovery and RSD for each analyte. Perform prior to implementation or when measurement system changes significantly.
Precision & Bias: non-standardized methods	Analyze QC sample in triplicate at three concentrations, the LOQ, mid-range, and upper-range. Analyze a method blank with each replicate set. Use a CRM for the mid-range QC sample if available. Process as three sets of samples through the entire measurement system for each analyte of interest. Each set, covering the concentration range of interest, should be processed and assayed on separate days. Verify & document reliable qualitative identification of analytes. For each analyte, calculate the mean % recovery for each day, for each level over days, and for all nine samples. Calculate the relative std. deviation for each of the means obtained. Perform prior to implementation or when measurement system changes significantly.
Selectivity	Incorporate appropriate tests for selectivity in the method. The evaluation for selectivity is done as part of the evaluation of bias.

* Each of these elements must be evaluated prior to using a test method and when the measurement system changes significantly.

C.5.1 EVALUATION OF SENSITIVITY:

C.5.1.1 Limit of Detection:

The Limit of Detection (LOD) shall be established for every analyte in each sample-type for which data are to be reported. If an agency or program requirement is in place, it shall be followed. In other instances any procedure for establishing the LOD that exists in EPA regulations or guidance or in the peer-reviewed literature, may be used. When clients request quantitative data be reported to the LOD, then the validity of the detection limit determination must be demonstrated by qualitative identification of the analyte in a QC sample containing the analyte at no more than 2X the LOD.

The LOD must be determined each time there is a change in the test method that affects how the test is performed, or when a change in instrumentation occurs that affects the sensitivity of the analysis.

All sample processing steps of the test method shall be included in the determination of the LOD. Instrument detection limits (determinations made without all sample processing steps required by the method) are not acceptable substitutes for the above referenced determinations.

All procedures used must be documented. Documentation must include the sample-type. All supporting data must be retained.

An LOD study is not required for any component or property for which spiking solutions or quality control samples are not available, or otherwise inappropriate (e.g., pH).

C.5.1.2 Limit of Quantitation:

The minimum limit of quantitation (LOQ) shall be established for every analyte for which quantitative data are to be reported. The minimum level of quantitation is the lowest value for which unqualified quantitative data may be reported by the laboratory. When determining the LOQ, if client, regulatory agency, or other requirements are in place, those requirements shall be followed. In other instances, any procedure for establishing the LOQ may be used as long as the validity of the determination is confirmed by successful analysis of a QC sample containing the analytes of concern at or near the claimed LOQ. A successful analysis is one where the recovery of each analyte is within the established MQOs. This single analysis is not required if the bias and precision of the measurement system is evaluated at the LOQ as described in Section 5.2.2.

The level of the lowest calibration standard shall be approximately equivalent to the LOQ.

If project-specific MQOs have quantitation limit requirements greater than the LOQ, the laboratory may, alternatively, analyze a QC sample containing the analyte at the lowest concentration of concern, All sample processing steps of the analytical protocol shall be included in the determination of the LOQ.

An LOQ study is not required for any component or property for which spiking solutions or quality control samples are not available or otherwise inappropriate (e.g., pH).

C.5.2 Determination of Bias and Precision

If sample results are to be reported over a concentration range, the bias and precision of the method must be evaluated over the working range. If the objective of the sample analyses is to only demonstrate the presence or absence of an analyte at a specific concentration, or to establish whether or not the concentration is above or below a specified value, then this determination need not be completed.

C.5.2.1 Standardized Methods

The following approach can be used for standardized methods, i.e., methods of known and documented precision and bias published by an organization generally recognized as competent to do so, where the client uses these MQOs. The approach in section C.5.2.2 below is required for modifications to standardized methods, laboratory-developed methods, or methods published in the scientific literature.

For each method and sample-type, the laboratory must analyze four replicate QC samples containing each analyte at 2-5 times the LOQ, or as otherwise stated in the method. The samples must be processed through all sample preparation and analysis steps in the method. The percent recovery and relative standard deviation must be calculated and these values compared to the MQOs of the method. If MQOs are not published in the method, the mean recovery and standard deviation are used to establish the laboratory-derived MQCs.

C.5.2.2 Non-standardized methods

This approach can be used for any method but is required for modifications to standardized methods, laboratory-developed methods, or methods published in the scientific literature. This approach is also required if a client provides MQOs that are different from those published in standardized methods. This approach may be used by the laboratory to document the performance of the method over the concentration range of interest for standardized methods if the laboratory chooses to perform this study.

Analyze QC samples in triplicate containing the analyte at or near the quantitation limit, at the upper-range of the calibration (upper 20%) and at a mid-range concentration. If a Certified Reference Material

(CRM) of the same matrix type as the samples is available, substitute the CRM for the appropriate replicates. Process these samples as three sets of samples through the entire measurement system for each analyte of interest. Each day one QC sample at each concentration is analyzed. A separate method blank shall be subjected to the analytical method along with the QC samples on each of the three days. (Note that the three samples at the LOQ concentration demonstrate sensitivity as well.) For each analyte, calculate the mean recovery for each day, for each level over days, and for all nine samples. Calculate the relative standard deviation for each of the separate means obtained.

Compare the results at each concentration to see if there is a significant difference in either bias or precision as a function of concentration. If there is no significant difference, calculate the mean recovery and standard deviation over the range of interest by combining all values. If there is a significant difference, calculate the mean recovery and standard deviation at each concentration. Evaluate the blank data for an indication of a positive bias. Compare these calculated results to the MQOs and determine if the method is adequate for its intended use. If no MQOs exist, the laboratory shall use these data to establish the MQCs.

C.5.3 Selectivity

The minimum requirement is to ensure that the measurement system is adequately selective. Appropriate selectivity checks established within the method should be followed, including mass spectral tuning, second column confirmation, ICP inter-element interference checks, chromatography retention time windows and related activities.

Appendix D.1
ONGOING QUALITY CONTROL REQUIREMENTS FOR DEMONSTRATION OF MEASUREMENT SYSTEM PERFORMANCE FOR CHEMICAL TESTING

This Appendix describes the minimum quality control (QC) procedures that are needed in order to document the quality of data obtained and to demonstrate that the laboratory was functioning in control. These requirements apply to virtually all types of testing (exceptions are noted). The laboratory shall ensure that the essential requirements in this Appendix are incorporated into their method manuals and/or the Laboratory Quality Assurance Plan. In addition to these minimum requirements, the laboratory shall also perform any additional procedures that are called for in the particular method that is being used. The laboratory shall have procedures for the development of acceptance/rejection criteria for the results of the quality control tests where no method or regulatory criteria exists. These criteria shall be based on the MQCs determined in the initial measurement system evaluation and shall be updated periodically based on the results from the analysis of QC samples.

The minimum quality control requirements are summarized in Table D-1

Table D-1

Evaluation Element	Section	Quality Control
System Cleanliness	D.1.2	Method blank
Calibration	D.1.3	Verification check or another calibration, second source Standard

Analytical System Performance	D.1.4	Laboratory Control Sample (LCS)
Analyst Proficiency	D.1.5	Annual performance verification
Limit of Detection	D.1.6	Spike sample at no more than 2X LOD. (when data are reported to LOD)
Limit of Quantitation	D.1.7	Spike at LOQ
Bias	D.1.8	Matrix spike/matrix spike duplicates *
Precision	D.1.8	Matrix spike/matrix spike duplicates , or replicate samples for each batch*
Selectivity	D.1.9	Confirm analyte identity; Confirm that quantitative results do not have a positive bias

* This QC test is not required in all applications.

D.1.1 Introduction

All measurement systems must be evaluated prior to their use on actual samples to determine measurement quality characteristics (MQCs) in representative sample-types (See Appendix C). The ongoing QC requirements established in this Appendix shall also be performed. Also, as part of the laboratory's training program, all laboratory staff involved in the analysis of a sample shall have passed a Demonstration of Analyst Proficiency prior to the analysis of any samples. The Demonstration shall be repeated whenever there is a significant change in instrument type, personnel, matrix or test method.

The results of these ongoing QC sample analyses shall be documented and reported and/or available along with the analytical results.

D.1.2 System Cleanliness

A critical component of analytical quality control is making certain that the species or properties whose level has been measured are not artifacts of the measurement system. Such artifacts can be caused by contamination of the instruments, the reagents, the preparation glassware, etc. Method blanks are used to assess the potential contribution of such contamination to the analytical results.

A method blank is used to assess measurement system cleanliness on each preparation batch for possible contamination during the preparation and processing steps. The method blank shall consist of a sample-type that is representative of the associated samples and is known to be free of the analytes of interest. The method blank shall be processed along with and under the same conditions as the associated samples to include all steps of the analytical procedure as if an actual sample was being analyzed. Any samples associated with (same batch) a contaminated method blank shall be reprocessed for analysis or the results reported with appropriate qualification. In those instances for which no separate preparation method is used (example: volatiles in water) the batch shall be defined as environmental samples that are analyzed together with the same method and personnel, using the same lots of reagents, not to exceed the analysis of 20 environmental samples.

While the goal is to have no detectable contaminants, each method blank must be critically evaluated as to the nature of the interference and the affect on the analysis of each sample within the batch. For purposes of taking corrective action a method blank is considered contaminated if the concentration of a reported analyte in the blank exceeds the greater of:

1. the established LOQ;

2. 1/10 of the measured concentration, which is above the LOQ, in any sample in the associated batch; or

3. 1/10 of the specified regulatory limit, which is above the LOQ, in any sample in the associated batch

Also, a method blank is considered to be contaminated if detected analytes otherwise affect the sample results as per the test method requirements or client MQOs.

If a method blank is determined to be contaminated, the source must be investigated and measures taken to minimize or eliminate the problem. Samples associated with a contaminated blank shall be evaluated as to the best corrective action for the affected samples (e.g. reprocessing or data qualifying codes). In all cases the corrective action shall be documented.

D.1.3 Calibration and Calibration Verification

Each day that analyses are to be performed using a particular instrument, the calibration of the instrument must be verified. See Section 5.5.4.3.3.2 for details.

D.1.4 Analytical System Performance

During routine use of a test method it is important to ensure that the analytical system is operating as expected (i.e., the performance of the system is in conformance with the expectation established by the initial measurement system evaluation). To document that the system is meeting expectations (and to identify if any problems are developing) a Laboratory Control Sample (LCS) is analyzed, including all steps of sample preparation and analyses, along with each batch of samples.

The LCS is used to evaluate the performance of the total analytical system, including all preparation and analysis steps. Results of the LCS are compared to established criteria and, if found to be outside of these criteria, indicates that the analytical system is out of control. In addition, trends in the LCS from batch to batch may be used as an early warning indication that problems may be developing and permit corrective action to be taken before the system reaches an out of control state. Any affected samples associated with an out of control LCS shall be reprocessed for re-analysis or the results reported with appropriate qualification.

The LCS shall be analyzed at a minimum of 1 per preparation batch. Exceptions would be for those analytes for which no spiking solutions are available such as total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen or turbidity. In those instances for which no separate preparation method is used (example: volatiles in water) the batch shall be defined as environmental samples that are analyzed together with the same method and personnel, using the same lots of reagents.

The LCS is a controlled sample-type, known to be free of analytes of interest, spiked with known and verified concentrations of analytes. Alternatively the LCS may consist of a media containing known and verified concentrations of analytes, such as a Certified Reference Material. All spike concentrations should be within the calibration range of the methods. Ideally, the LCS should contain all reportable analytes. The following shall be used in choosing components for the spike mixtures:

The components to be spiked shall be those that are reported to the client, including any permit specified analytes or client requested analytes. Unless otherwise required by a mandated test method or the client, the laboratory shall prepare the LCS using the following guidelines:

a. For those components that interfere with an accurate assessment such as spiking simultaneously with technical chlordane, toxaphene and PCBs, the spike should be chosen that represents the chemistries and elution patterns of the components to be reported

b. For those test methods that have extremely long lists of analytes, a representative number may be chosen using the following criteria. However, the laboratory shall insure that all targeted components are included in the spike mixture over a 2 year period.

1. For methods that include 1-10 targets, spike all components;
2. For methods that include 11-20 targets, spike at least 10 or 80%, whichever is greater;
3. For methods with more than 20 targets, spike at least 16 components.

c. Spike components should be chosen to represent the chemistries of the components to be reported. For chromatographic methods, the entire elution range should be represented by spike components.

The results of the individual batch LCS are calculated in percent recovery (%R) where:

%R = (Observed Value/True Value)(100)

Each individual analyte LCS recovery is compared to the acceptance criteria as published in the mandated test method. Where there are no established criteria, either in the form of MQOs from the client or in mandated methods, the laboratory should refer to the measurement quality characteristics of the method determined as part of the Initial Method Evaluation (see App. C) in order to establish the limits.. For spike results outside MQOs or MQCs, corrective action should be documented or the data reported with appropriate data qualifying codes and explanation to the client.

An LCS that is determined to be within the MQOs effectively establishes that the analytical system is in control and validates system performance for the samples in the associated batch. Samples analyzed along with a LCS determined to be out of control (e.g., an LCS failure) shall be considered suspect and the samples reprocessed and re-analyzed or the data reported with appropriate qualification.

If a large number of analytes are in the LCS, then it becomes statistically likely that a few will be outside the control limits. This does not indicate that the system is out of control, and corrective action may not be necessary. In this situation, upper and lower marginal exceedance (ME) limits can be established to assist with the corrective action. If more analytes exceed the LCS control limits than is allowed, or if any one analyte exceeds the ME limits, then the LCS has failed. This marginal exceedance approach is relevant for methods with long lists of analytes. It will not apply to target analyte lists with fewer than 30 analytes. (Note: These ME limits may be established using the MQO process, or are based on 4 times the standard deviation obtained in the Initial Measurement System Evaluation.)

The number of allowable marginal exceedances is based on a probability of 0.9 that any given analyte will exceed its control limit and a probability of 9 out of 100 that the total number of exceedances for a LCS is outside the allowable value. Table D-2 presents the allowable number of marginal exceedances for a given number of analytes in the LCS.

TABLE D-2. NUMBER OF MARGINAL EXCEEDANCES IN LABORATORY CONTROL SAMPLES

Number of Analytes in LCS	Allowable Number of Marginal Exceedances
> 74	5
69-60	4
59-51	3
50-40	2
39-30	1
<30	0

Marginal exceedances must be sporadic (i. e., random). If the same analyte exceeds the LCS control limit repeatedly, that is an indication that the problem is systemic and something is wrong with the measurement system. The source of error should be located and the appropriate corrective action taken. Laboratories must monitor the application of the sporadic marginal exceedance allowance to the LCS results to ensure random behavior.

D.1.5 Analyst Proficiency

An important aspect of analytical quality control is to determine and document analyst proficiency (i.e., the competency of the analytical team to perform the specific tests that are being conducted). In addition to documentation of education and training, a critical quality control measure that shall also be conducted is periodic demonstration of proficiency (see Section 5.5.6.2.c4).

Analyst proficiency shall be demonstrated at least annually for each test method that the analyst/work cell is performing and shall be documented using the form in Appendix E.

D.1.6 Limit of Detection

When the client or regulation requires that quantitative data at the LOD of the measurement system be reported, the laboratory must document that the measurement system is achieving the particular detection limit that is being reported as part of the ongoing quality control process The demonstration (the LOD is determined using the procedures described in Appendix C) is performed by the analysis of a QC sample with each sample batch containing the analyte of concern at no greater than 2X the detection limit. This demonstration shall be conducted for each sample type and for each analyte of concern but need only be used when data at the LOD is to be reported quantitatively. The requirement for ongoing demonstration of LOD does not apply in cases where results reported below LOQ are appropriately qualified.

D.1.7 Limit of Quantitation

When the client or regulation requires that the presence or absence of an analyte at the LOQ of the measurement system be reported, then the laboratory must conduct the necessary quality control procedures to document that the measurement system is achieving the particular LOQ that is being reported. The procedure must include all isolation/concentration steps, and sufficient analyte concentration must be used to ensure a quantitative analyte determination. Attainment of the LOQ (the LOQ is determined using the procedures described in Appendix C) shall be verified by analysis of QC sample containing the analyte of concern at the LOQ. The percent recovery for each analyte shall be determined. The minimum frequency for ongoing evaluation of the LOQ is annually or when a new and unusual matrix is encountered.

Results are compared to the MQOs as published in mandated test methods or provided by the client, or as established in the Initial Measurement System Evaluation

D.1.8 Matrix-Specific Bias and Precision

An integral part of determining the quality of laboratory data is documenting that the measurement system is yielding data suitable for the intended purpose (i.e., the bias and precision of the analytical system meets the client MQOs). To demonstrate that the bias and precision of the measurement system met the MQOs, a matrix spike/matrix spike duplicate (MS/MSD) pair are analyzed.

The frequency of the analysis of MS/MSD may be determined as part of a systematic planning process (e.g. Data Quality Objectives or MQOs) or as specified by a mandated test method. It will therefore be necessary for the laboratory to communicate with clients to determine the clients' needs, to determine which samples constitute a similar matrix type, and to ensure that sufficient sample volume is collected to determine the uncertainty of associated measurement results on samples using the procedures described below. If neither the client nor the test method requires this activity, the procedure described below need not be performed.

The components to be spiked shall be as specified by the mandated test method, by applicable regulation, or by the client. However, all analytes for which quantitative results are to be reported shall be determined. In the event the list of analytes to be determined contain components whose simultaneous presence will interfere with making an accurate assessment (but which are not expected to actually be present simultaneously in actual samples), such as spiking simultaneously with technical chlordane, toxaphene and PCBs, the spike should be chosen that represents the chemistries and elution patterns of the components to be reported.

For those test methods that have extremely long lists of analytes, a representative number may be chosen, using the following criteria for choosing the number of analytes to be spiked. However, the laboratory shall insure that all targeted components are included in the spike mixture over a 2 year period.

a. For methods that include 1-10 targets, spike all components;

b. For methods that include 11-20 targets, spike at least 10 or 80%, whichever is greater;

c. For methods with more than 20 targets, spike at least 16 components. Spike components should be chosen to represent the chemistries of the components to be reported.

For chromatographic methods, the entire elution range should be represented by spike components.

The results from MS/MSD are primarily designed to assess the precision and accuracy of analytical results in a given matrix and are expressed as percent recovery (%R) and relative percent difference (RPD), where:

$$\text{Average \%R} = [(C \text{ (matrix spike)} - C \text{ (unspiked sample)}) + (C \text{ (matrix spike duplicate)} - C \text{ (unspiked sample)})]/(2)(\text{Amount of Spike}) \times 100$$

and

$$\text{Average \%RPD} = [(\%R \text{ (MS)} + \%R \text{ (MSD)}) / 2] \times 100\%$$

The RPD may be calculated using either the analyte concentration or the percent recovery. Although both approaches are used in practice, use of recoveries to calculate the RPD may result in a different value from that when concentrations are used.

Results are compared to established criteria. Where there are no established criteria, either in the form of MQOs from the client or in mandated methods, the laboratory should refer to the measurement quality characteristics determined as part of the Initial Measurement System Evaluation (see App. C) in order to establish the limits. For results outside MQOs or MQCs, corrective action should be documented or the data reported with appropriate qualification and explanation(s) to the client.

There are several alternatives to using the traditional MS/MSD approach. These include: use of surrogate spikes to measure bias and precision and analysis of replicate samples of the same material to demonstrate acceptable precision. Surrogates are materials that have similar analytical properties to the analytes of concern but which are not naturally found in environmental samples. Surrogates are used most often in organic chromatography test methods and are chosen to reflect the chemistries of the targeted components of the method. Added prior to sample preparation/extraction, they provide a measure of recovery for each individual sample matrix.

When the surrogate approach is employed, the recovery and precision of the surrogates is used as the measure of bias and precision as described above for the MS/MSD approach.

D.1.9 Selectivity

The minimum requirement is to ensure that the measurement system is adequately selective. This includes performing the appropriate instrument set-up and performance checks (e.g., ICP inter-element interference checks, MS tune, determination of chromatography retention time windows).

Confirmation shall be performed to verify the compound identification when positive results are detected on a sample from a location that has not been previously tested by the laboratory, or any positive results must be noted as unconfirmed. Such confirmations shall be performed on both elemental and organic analytes or when recommended by the test method. Confirmation is required unless stipulated by the client. All confirmations shall be documented.

ATTACHMENT G

PBMS PRESENTATION
QUALITY SYSTEMS COMMITTEE MEETING
JUNE 10, 2002

NELAC Implementation Model for PBMS

July 2002

Quality Systems Subcommittee for PBMS:

Lara Autry, USEPA/OAR; **David Friedman**, USEPA/ORD; **Harry Gearhart**, Dupont Inc.; **Ken Jackson**, NYS DOH; **Carl Kircher**, FL DOH; **Jon Kauffman**, Lancaster Labs.; **Sheila Meyers**, TNRCC; **Jerry Parr**, Catalyst; **Scott Siders**, IL EPA; **Bill Telliard**, USEPA/OW; **Dan Wagner**, METS Hometest; **Bob Wyeth**, STL

Rest in Peace

1987-2002

- Performance Based Method System
- Performance Based Measurement System
- Performance Based System

Rest in Peace

1987-2002

- Performance Based Method System
- Performance Based Measurement System
- Performance Based System

The proposed standard has no mention of any of the above!

Goals of the Standard-Preparation Effort

- Allow flexibility in use of methods
- Provide an appropriate level of control
- Minimum requirements to ensure data quality
- Clear guidance for laboratories and assessors

Essential Elements of the standard

- Applies to all methods
  - Tiering/grandfathering
- Selection and Use of Methods
- MQOs and MQCs
- Analyst Proficiency

Significant Changes from Version Presented at NELAC 7i

- Grandfathering
- Tiering
- Clarity

Grandfathering clause

- At a particular laboratory:

  Initial measurement system evaluation is not required for methods already in use at the time of adoption of this standard

  (Appendix C – C.2 Background)

Method Selection

- When use of a method is mandated by a regulatory agency, or is specified by the client, only that method may be used
- In other cases, alternative methods may be selected
- Basis for selection is Measurement Quality Objectives (MQOs)

Use of Test Methods

- Must demonstrate that the measurement system provides data consistent with the intended use
  - Acceptable calibration
  - Acceptable initial evaluation
  - Acceptable ongoing evaluation

Instrument Calibration

- All instruments are to be calibrated
- Calibration is verified periodically or repeated
- Flexibility in selection of calibration procedures

Measurement System Evaluation

- MQOs are the focus of evaluation
- Goal is to evaluate the measurement system
  - laboratory, operator, instrument, method
- Measurement Quality Characteristics (MQCs) are determined for:

  range, precision & bias, sensitivity, selectivity
- MQCs must meet MQOs
- Demonstrate that system is capable of providing data for intended purpose

Matrices and Sample Types

Matrix: Drinking Water; Non-Potable Water; Solid & Chemical Materials; Biol. Tissues; Air & Emissions

Sample Type:

1. Perform the evaluation on the most difficult sample-type; e.g., a highly polluted waste water – can then apply to "cleaner" waste-water and drinking water.

OR

1. Perform the evaluation on site-specific samples

Initial Measurement System Evaluation

AS USED in the lab doing the evaluation

Required for all methods:

- EPA-approved methods used verbatim (but "tiering")
- New methods
- Modified methods
- Additional analytes (usually)
- Additional sample-types

Tiering concept

- Precision and bias need not be demonstrated over the concentration range for “standardized” methods
  - 4 replicates at mid-range concentration required
- Standardized method is a method of known and documented precision and bias issued by an organization competent to do so
- All other initial evaluation elements required

Initial MSE: Standardized Methods

Detection/Quantitation Limits

- Appropriate LOD determination
- Calculation of LOQ; verified experimentally

Bias; Range; Precision

- At the mid-point : 4 replicate samples

Selectivity

- Incorporate appropriate method checks

Additional requirements if data reported to DL

Initial MSE: Non-Standardized Methods

Detection/Quantitation Limits

- Appropriate LOD determination
- Calculation of LOQ; verified experimentally

Bias; Range; Precision

- At the LOQ: 3 spiked matrix
- At the mid-point : 3 CRMs, if available
- At the UL: 3 spiked matrix

Selectivity

- Incorporate appropriate method checks

Additional requirements if data reported to DL

Ongoing Measurement System Evaluation

- Calibration verification
- Annual LOD/LOQ determination/verification
- Laboratory Control Samples
- MS/MSD, where applicable
- Method blank
- Demonstration of analyst proficiency
- Selectivity measures

Analyst Proficiency

- Multiple options exist
  - 4 LCS
  - 4 Replicates
  - IMSE
  - PT sample
  - Split samples
- Form in Appendix E

Summary of Draft

- Initial method evaluation used to:
  - Document that measurement system (lab + method) is capable of providing data (MQCs) fit for intended use (MQOs) in typical matrix
- On-going quality control used to:
  - Document performance of method on actual samples
  - Demonstrate laboratory control at time of analysis

Next Steps – Recommendations to the QS Committee

- Expand the subcommittee
- Solicit input for further modification of the standard
- Present the modified standard for discussion at NELAC 8i
- Incorporate the modified standard into Ch. 5
- Present the standard for vote at NELAC 9